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METHODS OF

ANALYSIS AND CHEMICAL CONTROL

FOR USE IN THE FACTORIES OF THE
CUBAN-AMERICAN SUGAR
COMPANY

GUAYABAL, SOLEDAD, PUNTA ALEGRE,
CUNAGUA, MORONICO,
MANATIQUIA.

FOREWORD BY
GUILFORD L. SPENCER

NEW YORK
THE CUBAN-AMERICAN SUGAR COMPANY
1907

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Reg. A. Jaffa
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ROBERT DRUMMOND COMPANY, PRINTERS, NEW YORK

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METHODS OF ANALYSIS AND CHEMICAL CONTROL

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INTRODUCTION

In order that the results obtained in several factories may be comparable, it is necessary that the methods of sampling and analysis in all be conducted by uniform methods. Different methods, notably in the analysis of low products, frequently give widely different results with the same material, rendering comparisons unsatisfactory.

It is the purpose to supply methods that are applicable in all the Company's factories, in obtaining the data required for the report sheets and permanent records. Methods for the routine control work are not given, as it is only essential that these supply figures for use in a single factory, and not for comparisons in a group of factories. Minor details of processes are omitted.

The description of methods is preceded by definitions of terms as used by this Company in its records and reports.

Definitions of Terms

Mill Extraction.—This expression indicates the quantity of juice extracted from the cane by the mills, and is expressed in percentage terms of the weight of the cane.

Saturation Water. Saturation.—The water sprayed upon the bagasse before regrinding is termed the "saturation," and is expressed in percentage terms of the weight of the cane.

Dilution.—The dilution water is that part of the saturation water which is expressed with the juice, and mixing with it reduces its density below that of normal juice. Since the saturation water extracts sucrose and other solids from the bagasse, only a part of that in the juice is indicated by the dilution. The percentage of dilution is calculated from the densities of the normal and diluted juices and is reduced to terms of the weight of the cane.

Sucrose Retained. Retention.—This expression is designed to indicate the efficiency of the manufacturing processes; it shows the percentage of the sucrose in the raw juice, expressed by the mills, that is obtained in the commercial sugar.

Raw Juice, Normal.—The true normal juice is the juice as it actually exists *in* the cane. The juice cannot be separated from the cane in this state. By long established usage, the juice obtained by milling, without the use of saturation water, is considered to be normal juice, and, further, when saturation is practiced the undiluted juice from the first crushing is assumed to be of very nearly the density of the true normal juice. It is customary to apply a factor, deduced from experimental data, to reduce the density of the juice from the first crushing to a basis of the juice

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that would be obtained from all the mills without saturation. The density estimated in this way and the purity coefficient of the diluted or mixed juice are used in calculating the analysis of the so-called normal juice. The expression "normal juice" is used in this sense in the Company's records, and this use, though not strictly correct, is sanctioned by long and very general usage in the cane-sugar industry.

Raw Juice, Diluted.—This expression indicates the mixed juices from all the mills, when saturation water is used.

Syrup.—The syrup is the purified juice after concentration; it is termed *meladura* in the Spanish-American factories.

First Massecuite.—The first massecuite is that produced largely or wholly from syrup or meladura.

Mixed Massecuite.—This massecuite is composed of a small quantity of syrup used as a nucleus or footing for the strike and molasses from a first massecuite This is also termed "second massecuite."

Third Massecuite.—This is a massecuite boiled as described above for a mixed strike, except using molasses from the latter, instead of from first massecuite. When the expression "mixed massecuite" is used, this massecuite is termed "second" instead of "third."

Molasses.—The molasses products are numbered to correspond with the massecuite from which produced. In certain of the Company's factories, the second is the final or commercial molasses, and in others this product is the third molasses. The molasses product of lowest purity should be reported as final molasses.

Circulation Water.—The water used in condensing the vapors from the vacuum evaporating apparatus.

Residual Juice in the Bagasse.—The juice left in the bagasse at the completion of the milling process is termed " residual juice."

Fiber (Marc) in the Cane.—The woody residue left after exhausting the cane with water in the laboratory tests is termed "fiber," or "marc."

Standardization of Special Apparatus

Hydrometers.—As the Brix hydrometer indicates percentages of sucrose by weight in solutions of the pure carbohydrate, it may readily be checked by the polariscope. Three sugar solutions should be prepared with distilled water and refined sugar and of densities within the range of the hydrometer scale. The percentages of sucrose should be determined in these solutions, using standardized instruments. The hydrometer, when floated in these solutions at a temperature of 17.5° C., should, if correct, indicate percentages agreeing with those ascertained by the polariscopic method. If the observations are made at other than 17.5° C., the readings must be corrected by the tables in current use. The errors of the hydrometer at the top, middle and bottom parts of its scale should be noted. This standardized hydrometer may be used in checking other hydrometers. The hydrometers should be numbered or bear other marks for identification. Diamond-ink is convenient for marking these instruments and other glassware.

The hydrometer may also be checked by means of a pyknometer. This method is not usually a convenient one in cane-sugar factories.

Volumetric Glassware.—The glassware should be thoroughly cleansed with a warm solution of chromic acid in sulphuric acid, followed by rinsing with distilled water. Select flasks having necks of small internal diameter, preferably about 12 mm. The flasks should be thoroughly dry and should remain inside the balance case a few minutes after wiping them, before weighing. Recently boiled distilled water, cooled

to the room temperature, is most convenient for checking purposes in the factory laboratory.

A small error is necessarily permissible in graduated ware. The error should be less than 0.05 cc. for a 100-cc. flask or 0.03 for a 100-cc. pipette, and less for smaller apparatus.

Standardizing a Flask.—Counterpoise the dry flask on the balance with a similar flask and small weights. Remove and fill the flask to the mark with the distilled water, using a pipette in filling, and avoid wetting the neck of the flask or carrying bubbles of air into it. This manipulation requires great care. For small flasks the analytical balance and accurate weights must be used. The following table shows the number of grams of water the 100-cc. flask should hold at various temperatures, as weighed with brass weights in the air:

WEIGHT OF WATER AT VARIOUS TEMPERATURES

T° C.	Wt. 100 cc. (Mohr cc.)	Wt. 100 cc. (True cc.)	*T*° C.	Wt. 100 cc. (Mohr cc.)	Wt. 100 cc. (True cc.)
	Grams.	Grams.		Grams.	Grams.
17.5	100	99.772	24	99.88	99.655
18	99.99	99.765	25	99.86	99.635
19	99.98	99.75	26	99.84	99.61
20	99.96	99.73	27	99.82	99.59
21	99.94	99.715	28	99.79	99.56
22	99.92	99.695	29	99.76	99.535
23	99.90	99.675	30	99.74	99.51

In the above table allowance is made for the expansion of the glass.

If, for example, the flask holds 99.9 grams of water at 23° C., its capacity is 100 cc. (Mohr) and it is correct for use with a normal weight of 26.048 grams.

A standardized pipette is convenient for use in checking flasks.

Many polariscopes are now graduated at a standard temperature of 20° C., and according to the specifications of the International Committee should be used with a normal weight of 26 grams and a flask graduated to hold 100 true or metrical cubic centimeters. The column in the table giving the weight of 100 true cc. is for use in graduating apparatus to true cubic centimeters.

Since the Company's factories are equipped with apparatus graduated to Mohr's cc., and with books of tables for use with this system, the true cubic centimeter graduated ware will be little used.

Standardizing Burettes and Pipettes.—Burettes and pipettes are standardized by weighing the quantity of water they deliver. A flask is used in making the weighings. The table of weights of water is also used in checking burettes, etc. A definite time should be allowed for the drainage of the burette and pipette in checking, and in practice they should be used under the conditions of standardizing.

The sucrose pipette is most conveniently checked by comparing the polarization of a solution measured with it, with a test of the same solution using the balance. The pipette is made to deliver 2 normal weights in order to divide the error of measurement by 2.

Polariscope and Accessories.—The polariscope should be so arranged that the light from windows or cross-lights do not interfere with the observations. Under no circumstances should the instrument be located in a small compartment with the lamp. In the sugar-house laboratory, the polariscope should preferably be placed

in a box, open at one end and from which the body of the observer himself cuts off a part of the light of the room. The lamp should be placed in a well-ventilated compartment outside the box, the light being passed through a small opening to the polariscope. A piece of plate glass should cover the opening to prevent currents of air from causing the flame of the lamp to flicker.

The polariscope should be clamped securely to the floor of the box. The flame of the lamp should always occupy the same position or should not be moved without rechecking the zero of the polariscope. With most polariscopes slight changes in the relative positions of the instrument and the source of light, or the intensity of the light, displace the zero, rendering resetting of the polariscope scale necessary.

The dark products obtained in the present methods of manufacture necessitate the use of an acetylene or electric lamp in the polariscopic work.

Having properly arranged the polariscope and lamp the zero point must be checked by a blank observation, preferably with a tube filled with water in the trough. The vernier must be moved with the adjusting key until the instrument indicates zero under the above conditions. After setting the vernier, a standard quartz plate, reading approximately 96°, should be substituted for the tube of water and an observation made. The deviation from the marked value of the plate should be noted and allowed for in all observations.

The graduations of these instruments have attained such perfection that errors are rarely found in the scales. The eyes of different observers may differ, hence each should make check observations on the quartz plate and apply the proper corrections in his analytical work.

The eye should be allowed time to adjust itself to the instrument and recover from fatigue between tests. An observation should not be made immediately after looking at a bright light.

The observation tubes should be compared with one another, using the same sugar solution. The cover glasses should be examined frequently, and if their surfaces are scratched or not parallel, or if they show optical activity, they must be rejected. Care must be observed that the caps do not press unduly on the cover glass.

Each laboratory should be provided with a standard quartz plate, normal weights, and thermometer. These should be used only in checking the apparatus and not in the analytical work.

Weights and Measurements

Weights and Measurements.—The chemist must assure himself that all weights and measures are of one system, *e.g.*, all Spanish or all English; if not, they should be reduced to one system before proceeding with the control calculations. The accuracy of all scales and the calibration of all measuring tanks should be verified by the chemist.

Measurement and Weight of the Juice.—Where the juice cannot be weighed, its weight must be deduced from its volume and density. Where practicable, the volume of the measuring tank should be determined by running water into it from an accurately calibrated tank. This is not usually practicable in large factories, hence the volume must usually be calculated from the averages of several measurements. An overflow pipe or other device should be provided to indicate when the tank is full.

The juice entrains a large quantity of air which increases its volume. This introduces an error for which a correction based upon experimental data must be made. The volume of air entrained varies with the temperature of the juice and also with other conditions.

In deducing the weight of the juice from its volume, the latter should be corrected for temperatures other than 17.5° C. Gerlach's table for the expansion of sugar solutions may be used for this purpose. The weight of a cubic foot of water at 17.5° C. is to be taken at 62.346 lbs., and that of the U. S. gallon, 8.3317 lbs.

When the measurement of the juice is effected at moderate temperatures, the measured volume, without temperature correction, may be used, the specific gravity of the juice at the temperature of the measurement being employed in calculating the weight of the unit volume.

Measurement of the Saturation Water.—Two tanks should be used in measuring the water, one to be filling while the other is emptying. The weight of the water is calculated from its volume.

Where, through accident or other cause, the volume of the water cannot be determined, it must be estimated from the dilution of the juice by dividing the percentage of dilution by 0.7. This is necessary in order to estimate the weight of the bagasse.

Filter Press-cake.—The cake should be weighed, but as this is not usually feasible, its weight may be estimated from that of several average cakes. The number of presses emptied should be recorded for use in calculating the total amount of press-cake.

Massecuites and Molasses.—Massecuites should be measured immediately they are discharged from the pans. This is necessary on account of the expansion this material undergoes in the crystallizers. These measurements are not usually necessary except preparatory to a "Run Report."

It is usually difficult to accurately measure final molasses. This is due to the air it entrains. Where the molasses is slightly reduced with water and steam to bring it to a uniform density, it may be measured before pumping it from the factory, or afterwards in the large storage tanks. Measurements of very heavy molasses in tank-cars are unsatisfactory except where the measurement is made several hours after filling the tanks.

For control purposes, especially for the final report of the Crop, the actual weight of the molasses should be reported, though, in order not to delay the reports, a small quantity may be estimated at the close of each Run. The tank-cars should be tared each trip because often the buyer does not completely empty them.

Where measurements are necessarily made of heavy molasses in large tanks or tank-cars, corrections, determined by experiment, will be required for the entrained air. Heavy molasses will often entrain sufficient air to reduce its weight per gallon nearly one pound.

Sampling and Averaging

Preliminary Remarks.—Sampling should be so conducted that the quantity of material drawn from each tank, etc., bears the same relation to the total quantity. For example, having two equal tanks of syrup, one full, the other half-full, half as much should be drawn from the one as from the other, that the subsamples when mixed may represent all the syrup. This principle should be applied in all sampling.

In averaging analytical data, each analysis should be weighted by the quantity of material it represents.

Juice.—The ideal sample would be one drawn continuously in proportion to the quantity of juice flowing from the mills. Where the arrangement of the mills permits, a continuous or "drip" sample should be drawn from the canal leading from the crusher and first mill. This sample should be drawn throughout the sugar-house day, preserving it with formaldehyde or mercuric chloride, 1 part of the salt to

5000 of juice. It is for use in estimating the density of the normal juice. Where a continuous sample cannot be obtained, small, equal-measured subsamples should be drawn at least hourly, and united to form a sample representing the day's work. When not macerating, this sample may be dispensed with.

The mixed or diluted juice should also be sampled as it flows into the measuring tanks. A definite number of small cupfuls should be drawn per tank, and this number should not be changed during a sampling period, which is preferably the sugar-house day.

Duplicate samples of the mixed or diluted juice should be drawn, one for the sucrose and the other for density tests. One sample may be preserved with Horne's anhydrous subacetate of lead for analysis by his method. The least quantity of the lead salt that will insure preservation of the juice should be used. [Usually about 12 grams of the dry salt per litre of juice are required.] The duplicate sample, for the density test, may be preserved with either formaldehyde or mercuric chloride. It is optional with the chemist to draw a single sample for both sucrose and density tests, using 1 part of mercuric chloride to 5000 parts of juice as a preservative.

The preservation of samples must be carefully supervised. Only sterilized, wide-mouthed jars with stoppers should be used for storage purposes. Funnels should not be used to direct the sample into the jar. The small sampling cups should be thoroughly rinsed with the juice before drawing the subsamples.

Bagasse.—Subsamples should include all of the bagasse on one or more slats of the conductor. Composite samples, well protected from the air, may be preserved during a few hours with formaldehyde, but it is advisable not to extend the storage period to more than six hours. Clean galvanized-iron cans are to be used for storing the samples.

Filter Press-cake.—Subsamples should be cut from a definite number of cakes in each press by means of a brass cork-borer. The small samples may be collected during six hours without recourse to a preservative. Care should be observed that the samples be drawn from representative parts of the press-cakes.

Syrup and Molasses.—Syrup and thin molasses, to be reboiled, may be sampled in the storage tanks. Heavy commercial molasses should be sampled at frequent regular intervals at the pump-tank, forming a composite sample, representing the day's work. When shipping molasses, samples should be drawn from each tank-car, to form a composite sample representing the day's shipment. Composite samples should be prepared from the daily samples, for analysis to supply the data required in the Run Reports.

Sugars.—One hundred per cent of the packages should be sampled. As the truck passes onto the scale, a spoonful of sugar should be withdrawn and thrown into a tin-lined box, provided with a small funnel or hopper-like opening. Sugar already packed should be sampled with a trier.

Miscellaneous.—The principles described apply in all sampling and averaging in the control of the factory. Where practicable, composite samples of juices, massecuites, molasses, etc., should be prepared to supply data for the permanent records and daily reports, so that but a single analysis need be made daily of each class of material for these purposes.

Methods of Analysis

Sugar Cane.—Owing to the nature of the cane it cannot be accurately sampled without undue expense for appliances and labor. For this reason, and not on account of analytical difficulties, its analysis should be ascertained indirectly as follows:

The per cent sucrose in the cane = (weight of sucrose in the raw juice + weight of sucrose in the bagasse) ÷ weight of the cane × 100.

The per cent fiber, or marc, in the cane = fiber % bagasse × bagasse % cane ÷ 100.

The bagasse per cent cane is calculated as directed under Miscellaneous Calculations.

Juice.—*Brix.*—Strain the sample, through fine centrifugal lining, into a tall cylinder, letting the latter overflow. After an interval of about 10 minutes insert the hydrometer, at the same time blowing the foam from the surface of the liquid. After the instrument reaches the temperature of the juice, note the graduation on its stem on a level with the surface of the juice in the cylinder, and enter this number as the observed degree Brix. The temperature of the juice should also be noted and a correction be made, if it varies from 17.5° C., by Gerlach's table.

Sucrose.—Thoroughly mix the sample of juice that has been preserved with anhydrous subacetate of lead and filter a portion of it. Polarize the filtrate and calculate the percentage of sucrose, using Schmitz's table. If the Schmitz table providing for 10 per cent dilution is employed, divide the polariscope reading by 1.1. A copy of Schmitz's table, for use without dilution, will be supplied to each of the Company's laboratories, thus obviating the division by 1.1.

In case mercuric chloride has been used as a preservative, the analysis may be made by Horne's dry subacetate-of-lead method, or using subacetate in solution.

The estimation of the sucrose by double polarization is described under **Clerget Tests.**

Glucose.—(1) Using the composite sample preserved with anhydrous subacetate of lead: Mix the sample thoroughly and without previous filtration add powdered dry hydrosulphite of sodium (B.A.S.F.) in slight excess, *i.e.*, to faint acidity, and filter immediately; to the filtrate add sufficient anhydrous carbonate of sodium to produce a clear liquid on refiltration. Use this last filtrate in determining the glucose by Violette's or other standard method.

(2) Using a composite sample preserved with mercuric chloride: To a measured portion of the sample add a solution of normal acetate of lead for clarification, dilute to a definite volume, and filter. Add anhydrous carbonate of sodium to the filtrate in slight excess to precipitate the lead, and again filter. Determine the glucose in the filtrate by a standard method.

Solids by Drying.—*See* **Methods of Drying.**

Ash.—*See* **Ash Determinations.**

Bagasse.—*Sucrose.*—Digest 50 grams of small fragments of bagasse with 450 cc. of water and 5 cc. of a 5% solution of sodium carbonate, during one hour, at boiling temperature. A large flask, tared and fitted with a reflux condenser, should be used in making this test. A long small-bore glass tube will answer as a condenser.

After the digestion, cool and weigh the flask and contents. Drain off a portion of the solution, clarify it with subacetate of lead, and polarize, using a 400-mm. observation tube. The method of calculating the percentage of sucrose in the bagasse is illustrated by the following example, in which the fiber in the bagasse is assumed to be 45%:

Weight of flask + bagasse and water	998.5 gr.
" " "	500 "
" " bagasse and water	498.5 "
" " fiber in bagasse = 50 × 0.45 =	22.5 "
" " thin juice	476.0 "

Polarization of the thin juice (Horne's method) in a 400-mm. tube = 4.4. Dividing this number by 2 (on account of double-length tube) and referring to Schmitz's table, the per cent sucrose in the thin juice is 0.57, and $476 \times .0057 = 2.71$ grams sucrose in 50 grams of bagasse. ∴ the per cent sucrose in the bagasse is 5.42.

Moisture.—Small shallow trays made of 80-mesh brass-wire gauze are convenient for use in the moisture determinations. These should hold 80–100 grams of bagasse and should be narrow enough for weighing on the sugar balance. The bagasse should be dried at the temperature of boiling water in a steam- or water-oven, or for prompt results at 105° C., until there is no further loss of weight, or until there is a gain after a short interval, the lowest weight being accepted. The dry bagasse absorbs moisture with extreme rapidity, therefore the weighings must be quickly made.

The bagasse should be prepared for analysis by chopping it. It is not advisable to attempt to reduce it to a very fine state of division on account of the long exposure to the air that would be necessary.

Residual Juice.—A sample of the juice flowing from the bagasse roll of the last mill corresponds so nearly with the residual juice that it may be accepted as such. A sample of this juice is to be drawn at the time of sampling the bagasse, and its coefficient of purity determined.

Fiber, or Marc.—The fiber may be estimated by direct test, also indirectly. In the direct estimation, 20 grams of bagasse are placed in a tared cylinder of 80- or 100-mesh brass-wire gauze. The cylinder is placed in warm distilled water below 75° C., and after 10 minutes the water is drained from the bagasse. A gauze disc placed on the bagasse keeps it from rising above the water in the cylinder. The soaking in warm water is repeated once and then the bagasse is extracted five times in boiling water. After thorough drainage the cylinder of bagasse is placed in the oven and dried. These manipulations require very little attention. The dry fiber must be protected from absorbing moisture from the air while cooling.

In the indirect estimation the calculations are made as follows: Divide the percentage of sucrose in the bagasse by the coefficient of purity of the residual juice, and multiply the quotient by 100; subtract this last product (the juice solids of the bagasse) from the percentage of dry matter in the bagasse, and the remainder is the percentage of fiber (marc).

The direct method is to be preferred since the one sample of bagasse is used for all the tests.

Filter Press-cake.—*Sucrose.*—Reduce the sample to a fine state of division. This is readily accomplished, provided the presswork is satisfactory, by cutting the sample in a large mortar with scissors. The scissors should be held with a handle in each hand.

Rub 50 grams of the press-cake in a mortar to a cream with water and wash it into a 200-cc. flask. Add sufficient subacetate of lead to clarify the solution and dilute it to the mark with water. Shake the flask and contents very thoroughly and filter. Polarize the filtrate, using a 200-mm. tube. The polariscope reading is the percentage of sucrose in the sample.

Moisture.—Dry 5 grams as directed under **Methods of Drying.**

Sugars.—*Sucrose.*—Wash 26.048 grams of the sugar from the weighing capsule into a narrow-necked flask with water. Dissolve the crystals, then add sufficient subacetate of lead solution to effect the clarification; mix the contents of the flask by a rotary motion, add about 5 cc. of alumina cream, and dilute the mixture to 100 cc. Mix thoroughly and pour the contents of the flask onto a filter, covering the funnel to prevent evaporation. Reject the first portion of the filtrate, which is

diluted with the moisture of the filter-paper. Polarize the filtrate, using a 200-mm. tube.

Only the most accurate flasks in the laboratory should be used in the sugar tests. The polariscope should be checked with the standard quartz plate before polarizing the sugar.

For the double polarizations *see* **Clerget Tests.**

Glucose.—The solutions of sugar for glucose tests should be clarified with normal acetate of lead. The lead should be precipitated from the filtrate with anhydrous sodium carbonate in slight excess or with a solution of potassium oxalate, making due allowance in the latter case for the dilution. The strength of the sugar solution should be 5 grams or a multiple of 5 grams per 100 cc., or 5 grams to a multiple of 100 cc. The glucose in the filtrate is determined by Violette's method or other modification of the Fehling method. If Violette's method is used the strength of the solution should be such as to give a burette reading of about 20. With the strength of solution indicated above, a table of reciprocals facilitates the calculations.

Ash.—*See* **Ash Determinations.**

Moisture.—*See* **Methods of Drying.**

Massecuites and Molasses.—*Brix.*—Dissolve a definite weight of the material in an equal weight of water and determine the corrected degree Brix of the solution; multiply this number by 2 to obtain the degree Brix of the material. This method should be used except in the control of the pan-boiling.

Sucrose, Direct Polarization.—Standardized flasks should be used in this test. Wash the normal weight of the solution used in the Brix determination (=0.5 normal weight of the original material) into a 100-cc. flask; add sufficient subacetate solution to secure a good clarification, complete the volume to 100 cc. with water, mix thoroughly, and filter. Measure 50 cc. of the filtrate in a 50–55-cc. flask and add sufficient diluted acetic acid to acidulate the solution, then fill the flask to the 55-cc. mark with water. This solution is usually light enough colored to polarize in a 200-mm. tube. If the color is too dark filter the solution through powdered bone-black, rejecting the first half of the filtrate. If preferred, about 0.5 gram of powdered dry bone-black may be shaken with the solution in the flask to decolorize it. Should the solution still be too dark to polarize in a 200-mm. tube, use the half-length tube. The polariscope reading must be increased one-tenth to compensate for the dilution, and the polarization multiplied by 2 to reduce it to terms of the original material.

Solutions of first massecuites may usually be clarified with normal acetate of lead solution, thus obviating the acidulation with acetic acid before polarizing.

For the method by double polarization *see* **Clerget Tests.**

Glucose.—Prepare the solution for testing and conduct the analysis as described for sugars. Owing to the high percentage of glucose in molasses, great care is necessary in making the test.

Ash.—*See* **Ash Determinations.**

Moisture.—*See* **Methods of Drying.**

Condensation and Waste Waters.—Frequent sucrose tests should be made in water from the condensers and tail-pipes. In very exceptional cases the water may contain sufficient sucrose for a polariscopic test. The sucrose content is usually very small and may be estimated with sufficient accuracy by the α-naphthol test. This test is made as follows: Add 5 drops of a 20% solution of α-naphthol in alcohol to 2 cc. of the water in a test-tube, then add 10 cc. of concentrated **sulphuric acid,** using a pipette to conduct the acid to the bottom of the tube. If sucrose is present a violet zone forms between the two liquids; in the presence of 0.1% of sucrose the

reaction is obscured by the darkening of the solution; with 0.01% sucrose the color zone is that of a very dark-red wine; with 0.001% sucrose the entire solution is colored. Impure water will sometimes impart a faint color in the above test in the absence of sucrose, but this is usually readily distinguished from the color produced by sugar.

Clerget Tests.—*Molasses Samples.*—In making these tests the directions given must be strictly complied with.

The *direct polarization* is to be made as described under **Massecuites and Molasses.** The temperature of the polarization should be noted and when practicable should be near 20° C. The solutions should be prepared at the temperature of the polarization.

The *invert polarization* should be made at the temperature of the direct, using a water-jacketed tube for the purpose. The use of this tube is also desirable in the direct polarization. If the tube is not fitted with desiccator caps, except in cold weather, it may be necessary to make both polarizations at a temperature a few degrees above 20° C. in order to avoid the deposition of moisture on the cover glasses.

It is advisable to dissolve 2.5 normal weights of the molasses in 500 cc. in making up the solution for the direct polarization, to insure sufficient material for preparing the invert solution.

Remove the lead from the solution used in the direct polarization, using a strong solution of potassium oxalate as the precipitant, and adding a little alumina cream, increasing the volume of the molasses solution 10%. Measure 75 cc. of the deleaded solution into a 100-cc. flask, add to it 5 cc. of hydrochloric acid, containing 38.8 per cent of the acid, and mix the contents of the flask by a circular motion. Place the flask in a water-bath heated to 70° C. and raise the temperature of the sugar solution to 67°–69° C. in two and a half to three minutes, and maintain this temperature until the total heating period reaches ten minutes. Remove the flask and place it in cold water, so as to quickly reduce its temperature.

A thermometer should be placed in the sugar solution during the inversion.

The inversion may be conducted at room-temperature if preferred. After the addition of the acid, set the flask aside in the laboratory for 24 hours, then prepare the solution for analysis.

After the inversion wash the solution from the thermometer into the flask. Cool the solution to the temperature at which the direct polarization was made and fill the flask with water to the mark. If this solution is very dark, filter small portions at a time through powdered bone-black, rejecting the first half of the filtrate, or shake it with one gram of dry bone-black in a fine powder; filter and polarize the solution.

Water of the temperature at which the direct polarization was made should be circulated through the jacket of the polariscope tube while making the observation. Several readings should be made and their average and the temperature of the solution in the tube be noted. With a sensitive polariscope it is usually easy to make these observations accurately to 0.05° on the cane-sugar scale. Great care is essential both in the matter of the observation and the temperature, as there is no analytical work in the sugar laboratory more liable to personal error than in the invert polarizations. The errors of observation are multiplied in the calculations.

The following formula is to be used in making the calculations:

$$\text{Per cent sucrose (Clerget)} = \frac{100S}{142.66 - \frac{t^\circ}{2}},$$

in which S (in sugar-house work) is the arithmetic sum of the direct and invert readings.

The calculations based upon the strength of solutions given in this article are illustrated by the following example:

Direct reading (uncorrected for dilution) = 12.1 at 24° C.
Invert " " " " = −6.55 at 23.7° C.
The direct reading corrected = $(12.1+1.21)\times 2 = 26.62$.
" invert " " = $(6.55+0.65)\div 0.75\times 2 = 19.2$.

$$\text{Per cent sucrose (Clerget)} = \frac{100(26.62+19.2)}{142.66-\frac{23.7}{2}} = 35.02.$$

Notes on Clerget Tests.—In the analysis of a sugar the half-normal weight of the material should be contained in 75 cc. of solution for inversion. Similar concentration as regards the sugars present should be used in tests of all materials.

The Company's Run Reports require the determination of the sucrose by the Clerget method in the juice, sugars, and final molasses. In the case of the juice, Clerget tests should be made at frequent intervals of composite samples of the mixed juices preserved with mercuric chloride, using normal acetate of lead for clarifying. This procedure is advisable on account of the precipitation of dextrose and levulose by the basic-lead salt. These analyses are for use in estimating the Clerget test of the average juice for the Run. If these tests indicate a certain increase or decrease in the sucrose, as compared with the direct polarization, the same percentage of change is to be applied to the average polarization of the raw juice to estimate its Clerget number.

Ash Determinations.—The sulphated-ash method is to be used in all ash determinations. In this method the material contained in a shallow platinum dish is saturated with pure sulphuric acid, then gently heated until intumescence ceases. The heat is now to be gradually increased and the carbon burned. A muffle heated to dull redness is the best device for ashing sugar products. Care must be exercised not to fuse the ash. The sulphuric acid must be in sufficient quantity to convert the mineral constituents of the material into sulphates. To reduce the sulphated ash to terms of the normal ash, deduct one-tenth of its weight.

The following approximate quantities of the sugar products and sulphuric acid should be used in each test: 2 grams sugar and 0.5 cc. acid; 10 grams juice and 1 cc. acid; 2 grams molasses and 1 cc. of acid.

Methods of Drying.—With the exception of the bagasse, fiber (marc), and the filter press-cake, all dryings are to be effected in a vacuum drying apparatus. If the laboratory's facilities admit, all materials should be dried in the vacuum oven.

The drying of the bagasse and fiber has already been described under **Bagasse.**

Juice, press-cake, sugar, and molasses should be dried in very shallow dishes. The lead caps for bottles, 3-inch size, are well adapted for this purpose. Drying should be continued until the sample ceases to lose weight.

As a result of many comparative tests by many chemists, the vacuum method has been shown to be the most reliable.

Estimation of the Products in Process of Manufacture

Stock-taking.—The laboratory records should include the dimensions and capacities of all tanks, pans, crystallizers, mixers, etc., in the factory. Each piece of apparatus should bear a suitable mark for identification.

At the close of a Run the chemist, accompanied by assistants, should rapidly pass through the factory and measure and sample all material in process. At the same time the head pan-boiler should indicate with chalk the depth of massecuite in each pan and note its character. Beginning two or three days before the close of a Run, the head crystallizer men should record the "inches out" for each crystallizer as soon as it is filled.

In measuring the juice and syrup in the tanks it is usually easier to measure the depth of the empty space from the top of the tank, or, in other words, the "inches out," rather than the depth of the liquor.

Samples should be drawn, representing each class of material in stock. The laboratory records will show the analyses of the massecuites, so these need not be sampled.

From these data the quantity of materials of all kinds in stock, at a given hour, may readily be calculated. All stock data and calculations should be entered in a book to facilitate the work of estimating and in checking errors. It is preferable that all the calculations be made in the book itself.

It is sometimes more convenient to make the stock estimates a little in advance or after the close of the Run, in order to take advantage of a shut-down. In the one case the cane ground from the hour of stock-taking to the end of the Run is carried as "stock," and the sugar it will produce is estimated on a basis of the yield for the rest of the period. In the other case the estimated yield from the cane ground from the end of the Run to the shut-down is to be deducted from the total.

Estimation of the Sugar in Process.—The sugar in process in the raw juice may be estimated on a basis of the "sucrose retained" in a previous Run, or if such data are not at hand, the "retention" may be estimated at 88%. As the quantity of raw juice in stock is small, an over- or under-estimate here will introduce a very small error. Likewise the yield from clarified juice may be estimated at a somewhat higher rate, and that from syrup at a still higher rate, as these materials have already undergone a part of the manufacturing process. When first massecuite is made without boiling in molasses, its sugar yield may be estimated upon the above basis.

When molasses is "boiled-in," the yield of sugar from the massecuites may be estimated by means of the following formula:

Let x = percentage yield of commercial sugar from the massecuite;
B = degree Brix of the massecuite;
P = polarization of the massecuite;
p = polarization of the commercial sugar;
S = percentage of solids in the commercial sugar;
M = coefficient of purity of the final molasses.

Then
$$x = \frac{100P - BM}{p - \frac{SM}{100}}.$$

This method of calculation gives the yield of sugar with sufficient accuracy for the Run reports. The results would be accurate if true Brix, polarizations, and purities could be used, provided no solids are removed from the molasses in the processes of manufacture other than in the sugars.

The above formula is also used in calculating the yield of sugar from the molasses.

It will be noted in these methods that the yield includes all of the sugar presumed to be available, as of one grade. If more classes of sugar than one are made, their

relative proportions must be ascertained from the experience of the factory and the calculated yield apportioned accordingly, figuring each class to the average polarization for that grade of sugar.

Estimation of the Molasses in Process.—The sum of the weights of sucrose in the sugars made and estimated, in the molasses made, and in the known losses, is deducted from the sucrose received in the juice to ascertain the sucrose in the molasses in process. As there are always unknown losses that can only be measured by difference, the sucrose as estimated above must be further reduced by the factory's average of such losses. The net weight of sucrose is calculated to terms of final molasses on a basis of the estimated average purity of that in process.

As the sucrose received in the juice is calculated on the basis of an analysis that varies but little if at all from a Clerget test of this raw material, evidently Clerget tests and the true purity of the molasses should be used in these calculations.

For the purpose of providing figures for comparison with the results of previous Crops, the direct polarizations should be used in calculating the "sucrose account" in the Run Reports.

Miscellaneous Calculations

Saturation.—Correct the measured volume of the water for expansion by the customary tables, reducing it to the corresponding volume at 4° C. The weight of a cubic foot of water at 4° C. is 62.427 lbs.

$$\text{Per cent saturation} = \frac{62.427 \times \text{cubic feet water at } 4^\circ \text{ C.} \times 100}{\text{Weight of cane in pounds}}.$$

Dilution.—(Brix of normal juice − Brix of diluted juice) ÷ Brix of normal juice × 100 = dilution % diluted juice;

Diluted juice extraction % cane × dilution % diluted juice ÷ 100 = dilution % cane.

Mill Extraction.—(1) When not saturating: Mill extraction % cane = weight of juice ÷ weight of cane × 100.

(2) Mill extraction when saturating: Mill extraction % cane = % mill extraction including dilution − dilution % cane.

Weight of diluted juice ÷ weight of cane × 100 = mill extraction including dilution % cane;

Mill extraction including dilution % cane × dilution % diluted juice ÷ 100 = dilution % cane.

There are two methods of estimating the weight of the juice from its volume after deducting for the entrained air and the milk of lime added to it:

(1) Correct for expansion or contraction of the juice for temperatures above or below 17.5° C. by Gerlach's table, showing the volumes of sugar solutions at different temperatures, or the correction may be determined experimentally. The weight of a cubic foot of water at 17.5° C., 62.348 lbs. multiplied by the specific gravity of the juice corresponding to its degree Brix at 17.5° C., gives the weight of a cubic foot of the juice.

(2) This method is illustrated in the following example:

Degree Brix of the juice at 17.5° C. = 20.0
Temperature of the measurement = 27.0

The degree Brix at 27° C., applying the number in Gerlach's table for the correction of readings on the Brix scale for temperature, is 20.0 − 0.65 = 19.35, and the

corresponding specific gravity is 1.08039. The weight of a cubic foot of juice at 17.5° C., 62.348 lbs. × 1.08039 = 67.36 lbs., the weight at 27° C.

It is always preferable to actually weigh the juice when the factory facilities permit.

Bagasse.—When saturation is not employed: Per cent bagasse = 100 − % mill extraction. When saturation is employed: Per cent bagasse = (100 + % saturation) − % dilute juice.

These per cents are in terms of the weight of the cane.

Efficiency of Saturation.—(1) Ascertain the weight of fiber, juice, and water entering and leaving the mills.

The juice remaining in the bagasse per cent juice entering the mills = a; the water remaining in the bagasse per cent water entering the mills = b; $100b \div a$ = efficiency coefficient of the saturation, *i.e.*, the degree to which the water has mixed with the juice in the cane. (After I. H. Morse.)

(2) The following method by E. E. Hartmann is preferable to (1) when more accurate results are desired:

Let A = bagasse from the second mill;
B = " " " third "
Diffusion water = the quantity of water, assuming complete diffusion with the juice in the bagasse, that would bring the percentage of sucrose in A to that in B.

Then

$$\frac{(\text{Lbs. sucrose in } A \times \text{juice \% } B) - (\text{sucrose \% } B \times \text{lbs. juice in } A)}{\text{Sucrose \% } B} = \text{diffusion water (lbs.)};$$

Coefficient of diffusion (coefficient of efficiency) = diffusion water (lbs.) ÷ saturation water (lbs.) × 100.

When double saturation is practised, *i.e.*, the bagasse from the first mill is saturated with the thin juice from the last mill, the diffusion water is calculated from the bagasse data of the second and third mills, and also from similar data of the first and second mills, and these numbers are added to ascertain the total diffusion water.

This method is applicable with any combination of mills.

Efficiency of Mill-work.—A number indicating the efficiency of mill-work and taking into account the fiber content of the cane, is derived by the following formula of Noël Deerr. The percentages refer to the analysis of the bagasse: % residual juice in the bagasse ÷ (% fiber × specific gravity of residual juice) = efficiency number.

Forms Suggested for Laboratory Records

The following forms are suggested for use in systematizing the calculations for records and reports. The width of the columns must be arranged to suit the work. Local conditions may necessitate a rearrangement of the ruling:

EXTRACTION DATA

Run No.....

(Left-hand page of blank book)

Dates	Cane		Saturation		Dilute Juice	
	For Day	To Date	For Day	To Date	For Day	To Date
	Tons	Tons	Tons	Tons	Tons	Tons
Brought forward			(Allow 17 lines	for entries)		
To date.....						
To end of previous Run						
For Run						

EXTRACTION DATA

(Right-hand page of blank book)

Bagasse		Extraction; Dilute Juice		Extraction; Normal Juice			
For Day	To Date	For Day	To Date	For Day		To Date	
Tons	Tons	Per Cent	Per Cent	Per Cent	Tons	Tons	Per cent
				(Allow for 17 lines)			

JUICE ANALYSIS

Run No.....

(Left-hand page of blank book)

Dates	Diluted Juice							
	For Day		To Date		For Day		To Date	
	Brix		Brix		Sucrose		Sucrose	
	Degree	Tons	Tons	Degree	%	Tons	Tons	%
Brought forward				(Allow for 17 lines)				
To date.....								
To end of previous Run								
For Run								

JUICE AND BAGASSE ANALYSES

(Right-hand page of blank book)

Diluted Juice					Normal Juice			Bagasse			
For Day		To Date		To Date	To Date			For Day		To Date	
Glucose		Glucose		Purity	Brix	% Sucrose	% Glucose	% Sucrose	Sucrose, Tons	Sucrose, Tons	% Sucrose
%	Tons	Tons	%								
				(Allow for 17 lines)							

SUGARS

Run No.....

(Left-hand page of blank book)

Dates	First Sugar					Second Sugar				
	For Day			To Date		For Day			To Date	
	Bags	Test	Sucrose-bags	Sucrose-bags	Test	Bags	Test	Sucrose-bags	Sucrose-bags	Test
Brought forward.....				(Allow for 17 lines)						
To date.....										
To end of previous Run.....										
For Run.....										

FILTER PRESS-CAKE

(Right-hand page of blank book)

Filter Press-cake

For Day				To Date		
% Sucrose	Presses Filled	Press-cake, Tons	Sucrose, Tons	Sucrose, Tons	% Sucrose	
			(Allow for 17 lines)			

The above forms provide for the calculations of weighted averages and for bringing all averages to date daily, as is desired in certain of the Company's factories. For example, in the calculation of the analysis of the diluted and the normal juices: Brix-tons÷diluted juice, tons×100=degree Brix of diluted juice and Brix-tons÷ normal juice, tons×100=degree Brix of the normal juice. These figures are to date or for the Run, according to the data used. The same weights for Brix, sucrose, and glucose are used for both the diluted and normal juice.

In the case of the sugars, the number of bags multiplied by their tests gives the "sucrose-bags," and the total sucrose-bags÷total number of bags=average test.

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